Fundamentals and Comparisons for Organic Sample Extract Evaporation

Zoe Grosser and Robert Johnson
Horizon Technology, Inc., Salem, NH USA

Abstract
Sample preparation is a key part of the analytical process, contributing to reproducibility and accuracy of the final results. Generally, sample preparation for organic analysis requires the analytes of interest to be first extracted from the matrix. Then cleanup of the extract may be required to remove interferences arising from the matrix. Water is removed during the drying step if it was introduced from the samples. Finally the extract is reduced in volume to accommodate the detection limits needed for the analysis and the ability of the instrument to accommodate a large-volume sample. The evaporation/concentration step can be achieved with various technologies, including heat, vacuum, and blow-down. We will examine the parameters that go into each of these choices and describe criteria to consider in matching the sample to the technique. Further, solvent recovery has become increasingly important as the number of samples analyzed and the size of individual laboratory locations has increased. The implications for solvent recovery based on the type of evaporation will be discussed.

Introduction
Sample usually consists of organic analytes of varying volatility in a solvent/reagent of known volatility. The key is to efficiently remove the solvent/reagent without losing more than an acceptable amount of the analyte.

Experimental
Evaporation Parameters:
<table>
<thead>
<tr>
<th>Parameter</th>
<th>Open System</th>
<th>Closed System</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sparging</td>
<td>Less</td>
<td>More</td>
</tr>
<tr>
<td>Heating Sample</td>
<td>Less</td>
<td>More</td>
</tr>
<tr>
<td>Vapor Removal</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>Vacuum</td>
<td>No</td>
<td>Yes</td>
</tr>
</tbody>
</table>

Solvent Recovery
Solvent recovery in a closed system is possible because the solvent will provide the bulk of the vapor. Solvent recovery when large volumes of sparge gas are used or water vapor is co-mingled is not possible. Environmentally friendly to recover solvent.

Results and Discussion

Kuderna Danish
- Used for many years
- Became the early standard for evaporation/concentration
- Uses heat
- Partially closed system
- Reflux action
- Various types of systems have been introduced

<table>
<thead>
<tr>
<th>Type</th>
<th>Heat</th>
<th>Stir</th>
<th>Gas</th>
<th>Vacuum</th>
<th>Closed</th>
<th>Open</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rotary Evaporator</td>
<td>Yes</td>
<td>No</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td>Closed</td>
</tr>
<tr>
<td>NIPH Evaporator</td>
<td>Yes</td>
<td>Yes</td>
<td>Fan</td>
<td>No</td>
<td>Yes</td>
<td>Open</td>
</tr>
<tr>
<td>DryVap System</td>
<td>Yes</td>
<td>Yes</td>
<td>Fan</td>
<td>No</td>
<td>Yes</td>
<td>Open</td>
</tr>
</tbody>
</table>

Considerations:
- Volume of samples
- Type of samples (more volatile content)
- Laboratory sample load
- Initial investment considerations

Conclusions
- Sample preparation is a key step in the analysis process
- Parameters for evaporation and their impact on analysis have been discussed
- Improvements in matching the sample to the evaporation device characteristics can help reduce variability and improve recovery
- Examples for choosing a system based on sample volume, types of analytes, sample load, and initial investment considerations gives guidance on both analytical and business considerations.